

PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

Dyed Textile Yarn and Fabrics Prepared therefrom

We, EASTMAN KODAK COMPANY, a Company organized under the Laws of the State of New Jersey, United States of America of 343 State Street, Rochester, New York 14650, United States of America (Assignees of WITT IRION LANGSTAFF and THOMAS LUTHER SHEALY, JR.) do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to dyeing textile fibres, particularly acrylic textile fibres, and to the preparation of high-bulk yarn, and fabrics.

In the manufacture of high-bulk knit and woven fabrics such as sweater fabrics, the common practice is to blend in a yarn two types of fibres, fibres stabilized against heat shrinkage and fibres undergoing substantial linear shrinkage upon heating. When yarns containing these fibres are dyed at the boil as is customary, the fibres differentially shrink and a yarn is obtained having characteristic high bulk and loft readily distinguished from other yarns prepared from non-shrinkable fibres.

The process has several disadvantages residing partly in the fact that since dyeing is carried out after the yarn is prepared, the yarns are obtained only in single colours. Yarns containing blends of different colours are thus not obtainable. Also, since many types of fibre such as polyester and acrylic fibres cannot be successfully dyed together and since the high-shrink fibre cannot be dyed prior to blending, it has not been possible to make high-bulk blend yarns of this type.

Attempts have been made to use tow dyed material, such as acrylic tow and subsequently to impart the heat-shrink property to the dyed fibres. However, the dyed fibres tend

to gradually lose their heat-shrink property on natural ageing at temperatures common in warehouses and knitting mills. More particularly, the processing of tow-dyed fibre has been unsatisfactory in yarn making. The process also has limitations in dyeing method.

According to the present invention, there is provided a process for preparing a composite yarn, which comprises imparting heat-shrinkability to fibres of a hydrophobic synthetic polymer, the heat-shrinkable fibres being capable of being dyed at a temperature below that which causes substantial shrinkage, and combining the heat-shrinkable fibres with heat-stable textile fibres into a yarn.

The heat-shrinkable fibres are dyed at a temperature below that which causes substantial shrinkage either before or after being combined into the yarn, and the yarn, before or after being made up into a fabric (including a pile fabric) is heated to cause shrinkage of the heat-shrinkable fibre without substantially affecting the heat-stable fibre.

When performing the present invention, fibres composed of hydrophobic synthetic fibre-forming polymers, particularly acrylic and modacrylic polymers, modified so as to be dyeable at low temperatures, are treated to impart heat-shrinkage properties thereto, for example by heating, drafting and cooling the fibres without relaxing. The so-formed heat-shrinkable fibres are dyed before or after combination into a composite yarn with the heat-stable fibres, for example in package form, at a temperature below that causing the fibres to shrink, for example, at temperatures below 160°F., preferably between 140°F. and 160°F. such as 150°F. Subsequent drying of the fibres should also be carried out at temperatures below that causing loss of heat shrinkage, for example at 180°F. to 220°F. preferably at about 190°F. The composite yarns thus obtained contain-

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ing heat-shrinkable and heat-stable fibres may be heat treated at a later stage in the production of high-bulk fabrics.

The staple heat-shrinkable and heat-stable fibres may be combined in any suitable manner such as in the cotton system employing the usual picking, carding, drawing, roving and spinning operations. The staple heat-shrinkable fibre may first be dyed and dried at temperatures below which shrinkage occurs before combining with the heat-stable fibres into yarn in the cotton system. Alternatively, the yarn containing colourless heat-shrinkable fibres and heat-stable fibres can be package dyed or skein dyed before forming into fabric as by knitting or weaving after which the fabric is heated to give high bulk. An alternative manner is to blend slivers of dyed or undyed heat-shrinkable fibres with slivers of heat-stable fibre by known methods to produce the heat-shrinkable yarn. While the yarn comprising heat-shrinkable and heat-stable fibre is primarily designed for dyeing at low temperatures prior to fabrication, if desired the fabric or garment can be dyed at a low temperature followed by bulking with heat or dyeing and bulking of the fabric can be carried out at one time by dyeing at the boil.

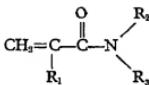
It should be noted that the dyeing of the unshrunk yarns from the spinners containing a mixture of colourless heat-shrinkable and heat-stable fibres, at the low temperatures required to retain the shrinkage properties, may not affect the heat-stable fibres since they may be dyable only at higher temperatures. However, fabrics containing the partially dyed yarn are pleasing in appearance. Alternatively, dyed heat-shrinkable fibres can be combined with heat-stable fibres which have been dyed in the normal manner at the boil and the two types of fibres spun into a lean yarn susceptible to bulking by means of heat. In preparing the high-bulk yarns, different deniers of the heat-stable and heat-shrinkable fibres can be, for example, from about 1 to 16 denier. In some cases, it may be necessary to use substantially different deniers for the two types of fibres to obtain the desired result.

Representative synthetic hydrophobic fibre-forming polymers capable of bearing the high heat-shrinkable property imparted thereto and which, in the shrinkable form, can be expected to be readily dyeable without substantial loss of shrinkage are, acrylonitrile homopolymers and copolymers including acrylonitrile-vinyl halide copolymers and acrylonitrile-vinylidene halide copolymers, polyolefins such as polypropylene, linear polyamides, e.g., nylon, linear polyesters such as the linear terephthalate polyesters, which polymers have been modified to improve their dyeability by blending or otherwise incorporating into the polymer alkylacrylamide polymers such as poly-N-alkylacrylamides, acrylic ester polymers including polyethylacrylate and polymethylmethacrylate, polyvinylpyridines, poly(vinylpyrrolidones), polyvinyl acetals, e.g., polyvinylbutyral, epoxy resins, e.g., epichlorohydrin-sulphide or epichlorohydrin-bis-phenol condensates, polyalkylimines, polycarbonates, polyoxalkenes, divalent metal salts of aliphatic carboxylic acids, and metal sulphonates.

Such heat-shrinkable polymers are usually capable of as much as 35 to 45% linear shrinkage, and on dyeing at the low temperatures contemplated by the invention can be expected to lose no more than about 2 to 10% of their heat-shrinkage property, so that dyed fibres and yarns are readily obtainable possessing as much as 30 to 40% retained shrinkage. The shrinkage of the fibre before or after dyeing may be determined by placing a given length in boiling water for two minutes followed by quenching with cold water and measuring the change in length. More accurate results may be obtained by using samples of tow in the test before cutting it into short staple fibre lengths. Yarn shrinkage is determined similarly except that the measurements of length of yarn before and after heating in water are made with a standard amount of tension per denier on the sample of yarn. Accordingly, by heat-shrinkable fibre or yarn, we mean fibre or yarn capable of 20% or more shrinkage on heating to elevated temperatures. An especially useful group of polymers to which substantial heat-shrinkability can be imparted which is retained through dyeing at relatively low temperatures, are the acrylonitrile polymers and copolymers containing at least 35% and up to 95% acrylonitrile units, and modified by, for example, 85-5% of vinyl pyridine units as described in U.S. patents 2,990,393 (Re. 25,533) and 3,014,008 (Re. 25,539) or modified by 65-5% of vinyl-pyrrolidone units, for example as described by U.S. patent 2,970,783, or modified with 65-5% acrylic ester or acrylamide units as described in British Patent Specifications Nos. 781,434, 781,435 and 781,436. Similar amounts of the other polymeric modifiers mentioned above are also useful. The polymers retain a useful amount of shrinkage under conditions that lead to significant dyeing of the fibres. A typical copolymer composition can be prepared as follows: 93 parts of acrylonitrile, 7 parts of 2-vinylpyridine, 1500 parts of water, 1.5 parts of ammonium persulphate, 1.5 parts of sodium metabisulphite, 10 parts of phosphoric acid, and 2.0 parts of sodium lauryl sulphate were heated at 40°C. for 15 hours. The resultant polymeric slurry, which had an intrinsic viscosity in dimethyl formamide of 1.4, was washed and dried, and after solution in dimethyl formamide, was spun under conditions that

gave a fibre having an appreciable degree of residual shrink. This material could be dyed under conditions that gave a dyed fibre that still retained about 70% of its original 5 shrinkage.

A preferred group of copolymers especially adapted to form heat-shrinkable fibres and which are readily dyeable at low temperatures without appreciable loss of shrinkage, 10 are modacrylic polymers composed of a mixture of (A) 70-95% by weight of a copolymer of from 30 to 65% by weight of vinylidene chloride or vinyl chloride and 70-35% by weight of acrylonitrile, and (B) 30-15% by weight of a second polymer from the group consisting of (1) homopolymers of acrylamidic monomers of the formula:



wherein R_1 is hydrogen or methyl, and R_2 and R_3 are each hydrogen or an alkyl group of 1 to 6 carbon atoms, (2) copolymers consisting of at least two of said acrylamidic monomers, and (3) copolymers consisting of at least 50% by weight of at least one of said acrylamidic monomers and not more than 50% by weight of a polymerizable mono-vinyl pyridine monomer.

A particularly efficacious group of modacrylic polymers for use in the low temperature dyeing process of the invention is an acetone soluble mixture of (A) 70-95% by weight of a copolymer of 30-65% by weight of vinylidene chloride and 70-35% by weight of acrylonitrile and (B) 30-5% by weight of an acrylamide homopolymer having the above formula wherein R_1 , R_2 and R_3 are as described above. The acrylamide homopolymer is preferably a lower N -alkyl-acrylamide polymer such as poly- N -methyl-acrylamide, poly- N -isopropylacrylamide and poly- N -tertiarybutylacrylamide.

As stated above, the heat-shrinkable fibres are combined into a yarn together with the heat-stable, (non-shrinkable) textile fibres. 45 After forming the yarn into a fabric, for example by knitting or weaving, heat is applied to differentially shrink the fibres to produce a high-bulk fabric. The method of applying heat to the fabric is not critical, for example, hot air at a temperature not adversely affecting the fibres and of the order of 250-300°F. can be employed. The increase in bulk is readily apparent from counting the increase in the number of courses and wales per inch and noting the increase in the weight of fabric per square yard. The improved fullness of hand will also be apparent. In this process, the combination of the specified shrinkable fibres and low temperature dyeing

produces fibres whose heat-shrink properties are maintained on natural ageing under room conditions over an extended period of time.

The heat-stable fibres which are used in the high-bulk or high-pile fabrics together with the shrinkable fibres may be any of a wide variety of fibres such as heat-stable modacrylic fibres described in British Patent Specification No. 806,875, cotton, mohair, wool, viscose, heat-stable acrylonitrile homopolymers and co-polymers such as those sold under the Registered Trade Marks Creslan, Acrilan, Dynel, Zefran, and Orlon, and linear terephthalate polyesters such as the linear cyclohexane - 1,4 - dimethanol terephthalate polyesters described in British Patent Specification No. 818,157 which have been heat stabilized as described in the patent. Heat-stabilized glycol terephthalate polyesters, including polyethylene terephthalate, described in the U.S. patent 2,465,319 are also very useful as the heat-stable component of high-pile fabric.

Dyes particularly useful for dyeing the heat-shrinkable fibres, particularly the modacrylic fibres described above include cationic or basic dyes well-known in the art for dyeing acrylic fibres, for example the Basacryl series of cationic dyes which are usefully used for dyeing acrylonitrile polymers such as Acrilan and Dynel at the boil, for example: Basacryl Yellow 5RL (C.I. Basic Yellow 25), Basacryl Red GL (C.I. Basic Red 29), Basacryl Blue 3RL (C.I. Basic Blue 53) and Basacryl Blue GL (C.I. Basic Blue 54); and the Sevron series of dyes including cationic cyanine, methine, anthraquinone, oxazine and triphenylmethane dyes such as Sevron Yellow L (C.I. Basic Yellow 13), Sevron Yellow R (C.I. Basic Yellow 11), Sevron Orange G (C.I. Basic Orange 21), Sevron Blue B (C.I. Basic Blue 21), Sevron Blue 2G (C.I. Basic Blue 22), Sevron Blue 5G (C.I. Basic Blue 4), Sevron Brilliant Red 4G (C.I. Basic Red 14) and Sevron Green B (C.I. Basic Green 3).

The following are representative of useful disperse dyes: 4-(2-methanesulphonyl-4'-nitrophenylazo) - N - β - cyanoethyl - N - β - acetoxyethylaniline; 4 - (6 - methanesulphonyl - 2 - benzothiozolylazo) - N - β - cyanoethyl - β - hydroxyethylaniline; 2 - nitro - 4 - N,N - dimethylsulphonamido - 4' - ethoxydiphenylamine; and 2 - nitro - 4 - sulphonanilidodiphenylamine. Representative of useful premetallized dyes are: Cibalan Yellow 2BRL, C. I. Acid Orange 87, Cibalan Red 2GL, C. I. Acid Red 211, Cibalan Orange RL, C. I. Acid Orange 88, Cibalan Blue BL, C. I. Acid Blue 168, Cibalan Brown 2GL, no C. I. number, and Cibalan Grey 2GL, C. I. Acid Black 62.

The usual stock of package dyeing methods can be used for dyeing the heat-shrinkable fibres. Dyeing assistants and levelling agents

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such as nonionic surfactants and phosphate compounds are useful as shown in the following examples.

Methods known in the art can be used for imparting the heat-shrink properties to the fibres. These methods include spinning the modacrylic fibres from solvent, passing the tow bundle over heated rolls and drafting the fibres to about 3 to 6 times their original length at a temperature of about 250-400° F. followed by cooling the fibres without relaxing or further heat treatment. A particularly useful method for imparting heat-shrinkage to the modacrylic fibres is to draft the fibres at a temperature of the order of 250-300° F. and spray with chilled water or pass them over a cool roll without relaxing or further heat treatment. When fibres such as the above modacrylic fibres are to be utilized as the heat-stable component of the high-bulk fabric, they are drafted with heat and relaxed and heat-stabilized in accordance with the usual practice.

The accompanying drawings, given by way of example, illustrate diagrammatically in Stage 1 the appearance of a composite plied yarn composed of heat-shrinkable fibres and heat-stable fibres, as obtained in a process such as the cotton process, the heat-shrinkable fibres having been dyed, before or after combining into the yarn, at a temperature below that causing heat-shrinkage. In Stage 2 the same yarn is shown diagrammatically substantially as it would appear after heating to cause differential shrinkage of the fibres and thus bulking of the yarn. Stage 1 of the drawings shows a plied yarn, each ply being a combination of heat-shrinkable and heat-stable fibres, but these are not essential features of the invention.

The following Examples illustrate the present invention.

EXAMPLE 1

A modacrylic fibre was prepared as described in British Patent Specification No. 806,875, comprising a mixture of a copolymer of vinylidene chloride and acrylonitrile and a minor amount of a poly(lower N-alkylacrylamide), the fibre being drafted with heat and cooled without relaxing to impart high heat-shrinkage thereto, after which the tow was cut to staple length. Fifty pounds of this 3-denier-per-filament, 1 1/2" length bright lustre, high shrinkage modacrylic staple fibre was dyed a brown shade in a Riggs and Lombard stock dyeing machine. The machine was filled to three-quarters capacity with water at 80°F. The fibre was loaded into the water by hand and distributed evenly. The machine was filled to the correct volume with water and circulation of the water was begun. The following chemicals were added to the machine and circulated for 10 minutes.

65 1.0% acetic acid (56%)

0.5% sodium acetate
1.0% fatty ester sulphate
1.5% self emulsifying organic phosphate
(Percentages based on fibre weight)

The following dyes (percentages based on the fibre weight) were pasted with acetic acid and dissolved in water, then added to the dye machine and circulated for 10 minutes.

70 0.36% Basacryl Blue GL

0.52% Basacryl Red GL

1.66% Basacryl Yellow 5RL

The dye bath temperature was raised from 80°F. to 140°F. over a period of 45 minutes. The fibre was dyed 60 minutes at 140°F. then rinsed at 120°F., removed from the machine and the water extracted in centrifuge. The fibre was then dried at 190°F.

75 Forty pounds of the dyed staple fibre was blended at the cotton picker with 60 pounds of a cyclohexane-1,4-dimethanol terephthalate polyester heat stable fibre of 4.5 denier-per-filament, bright lustre, 1 1/2" cut. Using conventional cotton spinning procedures, a 14/1 yarn with 9.8 TPIR singles was spun. This was two-plied with 4.28 twist and combed.

80 This yarn was knitted into a fabric on a Dubied NHF-5 V-bed knitter. It was 85 knitted from two cones of yarn. The greige fabric contained 6.5 courses/inch and 6 wales/inch and weighed about 7 ounces per square yard.

85 The greige fabric was then placed in a 90 boiling water bath for 20 minutes. It was removed and tumble dried at 220°F. The fabric showed a remarkable degree of bulking as evidenced by the change in construction to values of 10 courses/inch and 9 wales per inch and an increased weight to about 10 ounces per square yard. The fullness of hand of the finished fabric was remarkably improved as compared to the greige as-knit fabric. The finished fabric also had much improved cover. These improvements are attributed to the retained shrinkage ability of the stock dyed fibre, which caused the fibres to contract in the knit fabric and caused the polyester fibres to loop out from the yarn and fabric. The high resilience of the polyester fibre also contributed a springy feel to the surface of the fabric which was not lost in repeated washing and wear cycles.

100 Another way of demonstrating the bulking potential of the plied yarn containing the dyed shrinkable modacrylic fibre and the polyester fibres is to take a skein of the unknit yarn and measure its specific volume and size before and after treatment in boiling water.

| | Untreated Greige | Treated Boiled | 125 |
|--------------------------------|---------------------|-------------------|-----|
| Specific Volume Cu. In./Lb. | 64 | 106 | |
| Yarn Count, Cotton System | 13.8/2 | 10.2/2 | 130 |

The bulking potential of the yarn is not lost with age. This is shown by testing the contraction or shrinkage in length of the yarn over a period of time, illustrated by the following data obtained for a yarn similar to that used in the above example.

| | Shrinkage in Boiling Water |
|-------------------|-------------------------------|
| Freshly spun | 24.6% |
| Aged 11 days | 28.2% |
| Aged 3 months | 29.0% |
| Aged 3 1/2 months | 24.2% |
| Aged 4 months | 29.2% |
| Aged 4 1/2 months | 26.0% |

15 Thus the dyed shrinkable fibre or a yarn or fabric containing it can be shipped or stored for significant periods of time before it is bulked without loss of bulking potential. This is of considerable advantage as often the fibre is dyed in one plant, spun into yarn in a second plant, knit into a fabric in a third plant and made into a garment in a fourth plant and the final finishing of the garment may thus take place some time after the shrinking fibre is dyed.

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While in this example the heat-stable polyester fibre was not dyed, it can be stock dyed to any desired shade for cross dye or heather effects. Or, if desired, two or more 30 yarns can be made from different coloured stock dyed or natural staple fibres and used in knitting to produce bulky knit garments having stripe and other pattern colour effects as desired. Further, a natural yarn and a coloured yarn can be knit together into a fabric and bulked to produce a colour and white-striped or patterned sweater or dress at low cost.

EXAMPLE II

40 Example I was repeated using a heat-stable polyethylene terephthalate polyester fibre as the non-shrinking component in a blend with the shrinkable modacrylic fibre. A similar alteration in the bulk, hand and cover of the 45 finished fabric was noted.

EXAMPLE III

Example I was repeated using a heat-stable unmodified acrylonitrile polymer staple fibre as the non-shrinking fibre component of the 50 blend. When the knit fabric was finished, the same alteration in bulk as that found in Example I was noted.

EXAMPLE IV

A blend yarn was spun from 40% of the 55 3 denier-per-filament, 2 1/2" shrinkable modacrylic staple fibre and 60% of a cyclohexane-1,4-dimethanol terephthalate polyester staple fibre of 4.5 denier-per-filament and 2 1/2" length. Both fibres were bright lustre. The yarn was spun on the cotton

system into a 20/2 cotton count size yarn. The yarn was package dyed according to the following procedure:

1. Wind medium soft packages.
2. Set machine on five minutes—in and five minutes—out.
3. Add dyeing assistants over two cycles at 80°F. and run one cycle.
4. Add basic dyes over two cycles on outside—in portion at 80°F.
5. Raise temperature at 140°F. in 45 minutes.
6. Run one hour at 140°F.
7. Rinse, scour, rinse and dry at 190°F.

The dyed yarn was then knit into a fabric identified as O.G.—5 of 14 courses/inch and 13 wales/inch. The knit fabric was then steamed on a Hoffman press and the construction of the steamed fabric was again measured. The courses/inch increased to 17 and the wales to 14/inch, and the finished fabric exhibited a much fuller hand than the unsteamed, unbalked greige fabric.

The specific volume and size of the package dyed unknit yarn before and after bulking in a subsequent boiling water shrinking bath were as follows:

| | Treated Specific Volume Cu. In./Lb. | Treated Cotton Count, Yarn Size 10 | 90 |
|--|---|---------------------------------------|----|
| | | | 8 |

The shrinkage of this dyed yarn was 31% when exposed to boiling water immediately after dyeing. Three months after dyeing a second sample of the yarn was bulked in boiling water and was found to have 32% shrinkage.

The ability of the yarn of the invention represented by this example to be bulked after package dyeing is highly advantageous.

EXAMPLE V

Example IV was repeated except that the yarn was skein dyed according to the following procedure:

1. Wet out.
2. Use a cycle of 6 minutes forward and 4 minutes reverse flow on Hussong machine.
3. Add dyeing assistants at 80°F. and run one cycle.
4. Add one half of basic dye at 80°F. and run one cycle. Rotate skeins.
5. Add remaining one half of basic dye at 80°F. and run one cycle. Rotate skeins.
6. Raise temperature to 100°F. in one cycle. 115 Rotate skeins.
7. Raise temperature to 120°F. in one cycle. Rotate skeins.
8. Raise temperature to 140°F. in one cycle. Rotate skeins.
9. Raise temperature to 160°F. in one cycle. 120 Rotate skeins.

10. Run for one hour at 160°F. Rotate skeins every two cycles.
11. Rinse, scour, rinse, soften, extract, dry at 190°F.
- 5 The skein dyed yarn was knit into fabric and bulked with good results as in Example IV.

EXAMPLE VI

Example IV was repeated except that polyethylene terephthalate heat-stable fibre was substituted for the heat-stable polyester yarn of Example IV. The yarn was package dyed, knit and bulked by steaming. A similar alteration in the bulk of the finished fabric was noted.

EXAMPLE VII

Example IV was repeated except that an unmodified acrylonitrile polymer fibre was used as the heat-stable component of the blend. The knit fabric made from the package dyed yarn was steam treated to shrink the modacrylic component and a remarkable increase in bulk was noted.

EXAMPLE VIII

25 Example IV was repeated except that an apparel grade wool fibre was substituted for the heat-stable polyester fibre. Similar results were obtained as in Example IV.

EXAMPLE IX

30 Example IV was also repeated using cotton fibre in place of the heat-stable polyester fibre. Similar results were obtained as in Example IV.

While the invention has been illustrated 35 in Examples I to IX by the manufacture of knit fabrics, equally good results can be obtained in the manufacture of woven fabrics. For example, the package dyed unbulkled yarn can be woven into a loose weave fabric. The fabric is then treated with steam or boiling in a relaxed state so that the dyed shrinking component can cause contraction and a resultant bulking of the fabric to give it improved fullness of hand, thickness, heat insulation 40 ability and bulk.

45 It should be pointed that there is a particular advantage in using cyclohexane-1,4-dimethanol terephthalate heat-stable fibre in foregoing blends because of its low shrinkage 50 in boiling water of 0.5% and low hot air oven shrinkage of 4% at 220°C. By contrast, heat-stable polyethylene terephthalate polyester has 2.8% shrinkage in boiling water and 11.2% shrinkage in 190°C. hot air and some heat-stable acrylic fibres may have 3% or more shrinkage in boiling water and up to 20% shrinkage in 190°C. hot air. Since the bulking of a yarn composed of shrinking and non-shrinking fibres is based on the difference in potential shrinkage at a given temperature, it can be seen that with the same

shrinkable fibre, the cyclohexane-1,4-dimethanol terephthalate polyester fibre will make it possible to achieve higher bulk levels than other less heat-stable fibres which have higher shrinkage values in hot water or air.

EXAMPLE X

A staple yarn was spun from a blend of 40% of shrinkable modacrylic fibre such as used in Example I, 3 denier-per-filament, 2 1/2" cut, bright luster fibre and 60% of a cyclohexane-1,4-dimethanol terephthalate polyester staple fibre of 4.5 denier-per-filament and 2 1/2" cut of bright luster. The yarn was of a 1/42 ply cotton count construction.

The yarn was knit into a fabric containing 6.5 courses/inch and 6 wales/inch. The greige fabric was then dyed and bulked by the following procedure.

1. Bag fabrics or garments.
2. Wet out at 80°F.
3. Add assistants and circulate for 10 minutes at 80°F.
4. Add basic dyes and circulate for 10 minutes at 80°F.
5. Raise temperature to 160°F. in 45 minutes. Run one hour at 160°F.
6. Rinse, scour, rinse.
7. Bulk—Add 50% common salt and boil for 20 minutes. Cool slowly to 120°F. Rinse, soften, extract and dry.

The dyed and bulked fabric exhibited good bulk, fullness of hand and cover. It contained 12 courses/inch and 9 wales/inch.

EXAMPLE XI

Example X was repeated using in one case heat-stable acrylonitrile polymer fibre and in another case a heat-stable polyethylene terephthalate fibre in place of the heat-stable polyester of Example X. Similar bulking results were obtained.

EXAMPLE XII

A blend of 50%—3 denier-per-filament—2 1/2" bright shrinkable modacrylic fibre of Example I and 50% of the same fibre but non-shrinkable 16 denier-per-filament—2 1/2" bright fibre was spun into an 8/1 yarn and knit on a Tompkins circular knit machine into a flat knit fabric of about 28 ounces per square yard. Using low temperature dyeing techniques, the fabric was dyed according to the following procedure:

Dyeing Procedure

The fabric was dyed in rope form at 140°F. for one hour using the formula listed below to make a beige shade;

0.027% Basacryl Blue GL—C. I. Basic Blue 54 (Supplement)
0.033% Basacryl Red GL—C. I. Basic Red 29 (Supplement)

0.13% Basacryl Yellow 5RL—C. I. Basic
Yellow 25 (Supplement)

0.10% Direct Brown—C. I. 95.
0.0064% Superlitefast Blue 8 GLN

5 1.0% Acetic Acid (56%)
0.5% Sodium Acetate

40.0% Sodium Chloride
1.0% Fatty Ester Sulphate

1.5% Self-emulsifying organic phosphate
10 After dyeing, the fabric was rinsed and after treated with a cationic softer for 20 minutes at 120°F. To remove excess water, the fabric was vacuum extracted and dried at 190°F. The fabric was then passed through the normal pile fabric finishing operation of napping, shearing and heating at 280°F. to 300°F. to shrink the shrinkable fibre and provide a 2-pile height effect. The pile of the fabric was then heat-polished and 15 20 an imitation fur fabric resulted.

EXAMPLE XIII

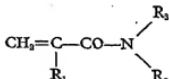
Using the yarn described in Example XII, a loop pile fabric with a 1/4" pile height was made on a 1/8" gauge tufting machine.

25 The resulting fabric was piece dyed as in Example XII. The fabric was finished in the same manner as Example XII to provide an imitation fur coat fabric.

Our copending Application No. 545/68 (Serial No. 1116926), divided from the present application, relates to a process of dyeing heat-shrinkable fibres of an acrylonitrile polymer, and to the use of such dyed fibres, together with heat-stable fibres, in the preparation of a high-pile fabric.

WHAT WE CLAIM IS:—

1. A process for preparing a composite yarn, which comprises imparting heat-shrinkability to fibres of a hydrophobic synthetic polymer, the heat-shrinkable fibres being capable of being dyed at a temperature below that which causes substantial shrinkage, and combining the heat-shrinkable fibres with heat-stable textile fibres into a yarn.
- 40 2. A process as claimed in Claim 1 in which the hydrophobic synthetic polymer is an acrylonitrile polymer.
3. A process as claimed in Claim 2 in which the acrylonitrile polymer contains from 35 to 95% of acrylonitrile units and from 65 to 5% of polymer units rendering the acrylonitrile polymer capable of being dyed at temperatures below that which causes substantial shrinkage of the polymer.
- 55 4. A process as claimed in Claim 2, in which the acrylonitrile polymer contains (A) 70 to 95% by weight of a copolymer containing 30 to 65% by weight of vinyl chloride or vinylidene chloride and 70 to 35% by weight of acrylonitrile, and (B) 30 to 5% by weight of a polymer which is either (1) a homopolymer of an acrylamidic monomer of the formula:



wherein R₁ is hydrogen or methyl, and R₂ and R₃ are each hydrogen or an alkyl group of from 1 to 6 carbon atoms, or (2) a copolymer of at least two acrylamidic monomers of the above formula, or (3) a copolymer consisting of at least 50% by weight of at least one acrylamidic monomer of the above formula and not more than 50% by weight of a polymerisable monovinyl pyridine monomer.

5 5. A process as claimed in Claim 4, in which the acrylonitrile polymer contains (A) 70 to 95% by weight of a copolymer of 30 to 65% by weight of vinylidene chloride and 70 to 35% by weight of acrylonitrile, and (B) 30 to 5% of poly-N-isopropylacrylamide.

6. A process as claimed in any preceding claim, in which the heat-stable textile fibres are fibres of a linear terephthalate polyester.

7. A process as claimed in Claim 6, in which the polyester is a 1,4-cyclohexanedimethanol terephthalate polyester or a glycol terephthalate polyester.

8. A process as claimed in any preceding claim, in which the heat-shrinkable fibres are dyed at a temperature below that which causes substantial shrinkage, before being combined into the composite yarn.

9. A process as claimed in Claim 8, in which the fibres are dyed at a temperature below 160°F.

10. A process as claimed in Claim 9, in which the dyed fibres are dried at a temperature between 180 and 220°F.

11. A process as claimed in any of Claims 8 to 10, in which the heat-shrinkable fibres, before dyeing are capable of being shrunk at least 20% by heating.

12. A process as claimed in Claim 11, in which the fibres are capable of being shrunk from 35 to 45% by heating.

13. A process as claimed in Claim 12, in which the fibres, after dyeing, are capable of being shrunk from 30 to 40% by heating.

14. A process as claimed in any of the preceding claims, in which the heat-shrinkable fibres are prepared by preparing fibres of the hydrophobic synthetic polymer and then heating, drafting and cooling the fibres without relaxing or further heat treatment.

15. A composite yarn whenever formed by the process of any of Claims 1 to 14.

16. A process of forming a high-bulk fabric which comprises forming a woven or knitted fabric from a composite yarn according to Claim 15, and heating the fabric to cause substantial shrinkage of the heat-shrinkable fibres.

17. A process as claimed in Claim 16, in

which the heating is carried out at a temperature in the range 250° to 300°F.

18. A high-bulk fabric whenever formed by the process of Claim 16 or 17.

5 19. A process of forming a high-pile fabric wherein a composite yarn according to Claim 15 is made up into a pile fabric and the pile fabric is heated to cause substantial shrinkage of the heat-shrinkable fibres.

10 20. A process as claimed in Claim 19 in

which the pile fabric is heated to a temperature in the range 250° to 300°F.

21. A high-pile fabric whenever made by the process of Claim 19 or 20.

22. A process for preparing a composite 15 yarn as claimed in Claim 1 and substantially as hereinbefore described.

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COMPLETE SPECIFICATION

1 SHEET

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